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Supporting Information

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Overcoming the Instability of Gaseous Peptide Phosphate Ester Groups by Dimetal Protection**

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Supplementary Information

Details of crystal structure determination for $[Ga_2(bpbp)(OH)_2(OH_2)_2](ClO_4)_3$ $(2(ClO_4)_3$

Addition of a large excess of tetraethylammonium perchlorate to a solution of Ga(NO3)3·6H2O (140 mg, 0.3496 mmol) and 5 mL MeOH containing Hbpbp (100 mg, 0.175 mmol) resulted in the formation of crystals suitable for single crystal X-ray diffraction.

Diffraction data were collected using a Bruker-Nonius X8 APEX-II instrument (Mo Kα radiation). Crystal structure data for $C_{90}H_{111}Cl_6Ga_4N_{19}O_{36}$: crystal size: 0.24 × 0.12 × 0.08 mm^3, monoclinic, crystal quality poor, *C*2/*c*, $a = 32.4564(18)$, $b = 1\ 1.9540(6)$, $c = 31.7882(18)$ Å, $\beta = 120.424(2)^o$, $V = 10635.0(10)$ $Å^3$, $Z = 4$, $\rho_{calcd} = 1.578$ gcm^{-3}, $2\Theta_{max} = 52.06^o$, Mo-K_α radiation, $T = 150$ K, $\mu = 1.245$ mm^{-1}. Refinement on F^2 for 10434 reflections and 663 parameters gave R_1 = 0.0701 and wR_2 = 0.1932 for data with $I > 2\sigma(I)$, R_1 = 0.0904, wR_2 = 0.2080 for all data; $-1.325 < \Delta\rho < 2.473$ and e$Å^3$. All non-H atoms were refined with anisotropic thermal displacement parameters, except O10-O18. H atoms bound to C were placed in calculated positions and refined using a riding model. O-bound H atoms were refined with O-H distances restrained to 0.84 Å and U_{iso}(H) = 1.5$U_{eq}(O)$. Structures were solved using SIR-92 and refined using SHELXL-97 operating under the WinGX software package. Two out of three ClO_4^- anions displayed unresolved disorder and the non-coordinating H_2O molecule was assigned an occupancy of 0.58. Structure deposited in the Cambridge Crystallographic data CCDC No XXXXXX.

Table S1. Selected bond lengths [Å] and angles [°] for (2$(ClO_4)_3$).

N(1)−Ga(1)	2.129(4)
N(2)−Ga(1)	2.090(4)
N(3)−Ga(1)	2.069(5)
N(4)−Ga(2)	2.114(4)
N(5)−Ga(2)	2.096(5)
N(6)−Ga(2)	2.055(4)
O(1)−Ga(1)	2.037(4)
O(1)−Ga(2)	2.049(4)
O(2)−Ga(1)	1.982(4)
O(3)−Ga(1)	1.862(4)
O(4)−Ga(2)	1.865(4)
O(5)−Ga(2)	1.988(4)
Ga(1)−Ga(2)	3.641(8)

O(3)−Ga(1)−O(2)	97.86(17)
O(3)−Ga(1)−O(1)	93.18(15)
O(2)−Ga(1)−O(1)	86.33(15)
O(3)−Ga(1)−N(3)	92.77(18)
O(2)−Ga(1)−N(3)	167.73(17)
O(1)−Ga(1)−N(3)	87.00(16)
O(3)−Ga(1)−N(2)	98.91(18)
O(2)−Ga(1)−N(2)	88.09(17)
O(1)−Ga(1)−N(2)	167.28(16)
N(3)−Ga(1)−N(2)	96.31(17)
O(3)−Ga(1)−N(1)	172.41(17)
O(2)−Ga(1)−N(1)	88.98(17)
O(1)−Ga(1)−N(1)	90.53(15)
N(3)−Ga(1)−N(1)	80.80(18)
N(2)−Ga(1)−N(1)	77.93(17)
O(4)−Ga(2)−O(5)	97.21(16)
O(4)−Ga(2)−O(1)	92.56(15)
O(5)−Ga(2)−O(1)	86.00(15)
O(4)−Ga(2)−N(6)	92.46(17)
O(5)−Ga(2)−N(6)	168.52(17)
O(1)−Ga(2)−N(6)	87.40(16)
O(4)−Ga(2)−N(5)	98.76(17)
O(5)−Ga(2)−N(5)	86.47(17)
O(1)−Ga(2)−N(5)	167.10(16)
N(6)−Ga(2)−N(5)	98.20(17)
O(4)−Ga(2)−N(4)	172.26(17)
O(5)−Ga(2)−N(4)	89.73(16)
O(1)−Ga(2)−N(4)	91.34(15)
N(6)−Ga(2)−N(4)	81.04(17)
N(5)−Ga(2)−N(4)	78.17(17)

Table S2. Hydrogen bonds [Å, º] in (2(ClO_4)$_3$),

D−H	d(D−H)	d(H..A)	<DHA	d(D..A)	A

O2−H1W	0.792	2.100	162.93	2.867	N9^{i}
O2−H2W	0.840	1.657	169.28	2.488	O4
O3−H3W	0.842	2.098	162.71	2.913	N7^{i}
O5−H4W	0.733	2.069	173.86	2.799	O7^{i}
O5−H5W	0.838	1.685	160.55	2.491	O3
O4−H6W	0.837	1.973	178.27	2.810	O9^{i}

Symmetry code: (i) -*x*+1, -*y*+1, -*z*

Table S3. Peptides not tagged in the presence of **1**.

Sequence	Tagged yes/no
AEFVEVTK	No
AFKDEDTQAMPFR	No
ALELFR	No
ASEDLKK	No
FKDLGEEHFK	No
GDVAFVK	No
HGLDNYR	No
HIQKEDVPSER	No
HLVDEPQNLIK	No
HQTVPQNTGGK	No
IPAVFK	No
KVPQVSTPTLVEVSR	No
LHDRNTYEK	No
LTEEEKNR	No
LTEWTSSNVMEER	No
NQETSEEYQIK	No
TPEVDDEALEK	No
VEQGASVDKR	No
YLYEIAR	No
YIcDNQDTISSK	No
VASLR	No
IIAEK	No
AVGNLR	No
LNFLK	No
TPVSEK	No
GLDIQK	No
VNELSK	No
VEQGASVDK	No
APNHAVVTR	No
DLGEEHFK	No
HQTVPQNTGGKNPDPWAK	No